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Penetration and Distribution of Calcium Ions in Thermal-Processed Apple Slices

SUMMARY—Stayman apple slices were treated in a solution containing calcium chloride ($CaCl_2$) and radioactive calcium ($Ca^{45}Cl_2$) to determine movement of the Ca ion into the tissue. Autoradiograms were developed from specimens prepared from Ca^{45}-treated slices. From the resultant photographic prints, penetration and distribution of Ca ions in apple tissue under different treatments were ascertained. Dipping or submerging the slices in Ca solution allowed only the surface to receive Ca. Processing slices in a Ca salt medium produces similar results. Use of a vacuum-pressure technique while the slices were submerged in the Ca solution provided a more satisfactory method for impregnating the tissue. Slices treated by this method showed essentially an even distribution of Ca ions in the tissue.

Radioactive Ca ions may be used to determine the efficiency of canning where plant tissues are treated with Ca salts.

INTRODUCTION

Numerous studies show that Ca salts reduce the tissue breakdown of different plant products caused by thermal processing (Collins and Wiley, 1963; Erickson and Boyden, 1947; Giggard, 1954; Kertesz *et al.*, 1940; Pyke and Johnson, 1940). Also, relatively soft and overripe fruits and vegetables, when processed with Ca salts, often yield a salable product that otherwise could not have been prepared (Archer, 1962; Collins and Wiley, 1963; Hills *et al.*, 1947; Stirton and Hills, 1950). Ca ions react with structural polysaccharides to form a "gel" which lends strength to the tissue (Kertesz *et al.*, 1940; Powers and Esselen, 1946). To obtain chemical and physical modifications, Ca ions must penetrate the tissues, thereby facilitating intimate contact between Ca ions and structural components. When Ca ions contact only surface tissue, this area becomes "hardened," but the inner tissue remains soft (Anon., 1947; Guadagni, 1950; Scott and Twigg, 1964).

Movement of salts into plant tissues has been studied to a limited extent. Pederson and Albury (1962) used indicator dyes to ascertain movement of brine into cucumbers. Giggard (1954) measured the Ca content at different depths of Ca-treated sweet potatoes to determine penetration into the roots. Penetration studies of various sugars into cherries for candying are presently being conducted with C^{14}-labeled sugars (Moser, 1965). Movement of Ca ions into apple slices has been estimated primarily by observing relative differences in firmness.

Several methods have been practiced involving treatment of apple slices with Ca salts. Common practices include dipping or soaking slices in a Ca salt solution. Attempts have been made to reduce softening by adding Ca salts to the can in which the product is processed (Anon., 1947; Holgate and Kertesz, 1948; Kertesz, 1947). None of these methods produce satisfactory results; "case-hardening" invariably occurs. A more satisfactory method for impregnating the tissue involves use of a vacuum-pressure technique while the slices are submerged in the Ca solution (Hills *et al.*, 1947; Stirton and Hills, 1950).

Even though the latter procedure is most effective, processors may still question the efficiency of this operation. Since slices with uniform firmness are desired, there must be an even distribution of Ca in the tissue (Holgate and Kertesz, 1948; USDA, 1953). With greater emphasis being placed on product texture, a more thorough understanding of Ca ion movement into processed plant tissues is needed.

This paper presents the procedures employed to determine depth of salt penetration and relative concentration of Ca ions in treated apple slices.

MATERIALS AND METHODS

Description of apples

The apples (Stayman var.) used were grown at the Agronomy-Horticulture Plant Research Farm and picked at 3 different stages of maturation. Storage was at 34°F.

Apples were harvested at immature, mature, and overmature stages of maturation. Fruit from the first 2 pickings was processed after 50 days of cold storage, and the overmature apples were used immediately after harvest.

Preparation of apples

On designated dates, conventional methods were employed to peel, core, and trim the fruit. Random samples of 100 g were obtained from apples sliced longitudinally into 8 equal sections.

Radioactive calcium test solutions

Solutions containing $CaCl_2 \cdot 2H_2O$ (calculated as 0.5% anhydrous $CaCl_2$) and 4 microcuries (μc) of Ca^{45} per 100 ml of test solution were used to treat 100 g of slices. Apple tissue treated in this concentration of Ca^{45} produced the subsequent autoradiograms in 10–14 days. A higher concentration of Ca^{45} shortens the time required to develop autoradiograms.

Treatment of slices

In most instances, slices were submerged in 500 ml of the radioactive solution. Slices, when deaerated, were placed under a partial vacuum of *ca.* 29 in. Hg and held for various periods. The vacuum was then broken with steam, and the pressure was brought to 10 psi and held for 2 min.

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Slices designated for further heat treatment were weighed into No. 1 cans and processed in boiling H_2O for 8 min.

Slices were treated with $Ca^{45}Cl_2$ prior to processing, during the vacuum operation, and during processing in the can.

Treatment prior to processing

Raw slices of mature apples were submerged in a $Ca^{45}Cl_2$ solution at 68°F and allowed to absorb the salt for 15 min. These slices were handled in 3 ways: submerged only, submerged and then processed; submerged, subjected to vacuum and steam, and then processed.

Treatment during vacuum operation

Slices of immature apples were submerged in a solution containing $Ca^{45}Cl_2$ and then subjected to vacuum for 0, 5, or 15 min. Slices were then steamed and processed. The overmature slices were deaerated for 0 or 15 min and then steamed and processed.

Treatment during processing

Mature slices were subjected to vacuum for 2 min. The vacuum was released and steaming was accomplished at 10 psi for 30 sec. Slices were not submerged in solution during deaeration and steaming. This operation may closely approximate a commercial process. The slices were then placed into a No. 1 can with a solution containing $Ca^{45}Cl_2$ and processed 8 min at 212°F.

Tissue section preparation

Transverse and longitudinal sections about 2 mm thick were cut from the central region of the slices. The sections were freeze-dried overnight and then placed on Kodak No-Screen Medical X-ray film. The film remained in contact with the specimen long enough to accumulate 2×10^6 beta particles per sq cm. The exposed film was developed in a dark-room with a safety light (safe-light filter, Wratten Series OC) located in the background.

Evaluation of calcium penetration and distribution

The autoradiogram negative was photographed to obtain a "positive" image. A print was made from the positive, showing the image in actual size (Boyd, 1955). Dark areas on the print indicated the presence of Ca^{45}.

RESULTS AND DISCUSSION

MATURE SLICES SUBMERGED in a solution of Ca salt did not receive even distribution of Ca (Fig. 1). In every autoradiogram prepared, little or no Ca penetrated the center tissue. When raw slices were submerged 15 min, Ca ions penetrated only 1 to 2 mm deep (*A*). When similarly prepared slices were processed (*B*), Ca moved somewhat deeper into the tissue. It appears that Ca becomes more evenly distributed when the submerged slices, subjected to deaeration and pressure, are processed (compare *C* with *B*).

Autoradiograms in Fig. 2 show the depth of Ca penetration into relatively immature apples in which the period of deaeration was progressively lengthened. When no vacuum was applied (*A*), Ca ions penetrated only the outer portion of the slices. In actuality this area would be

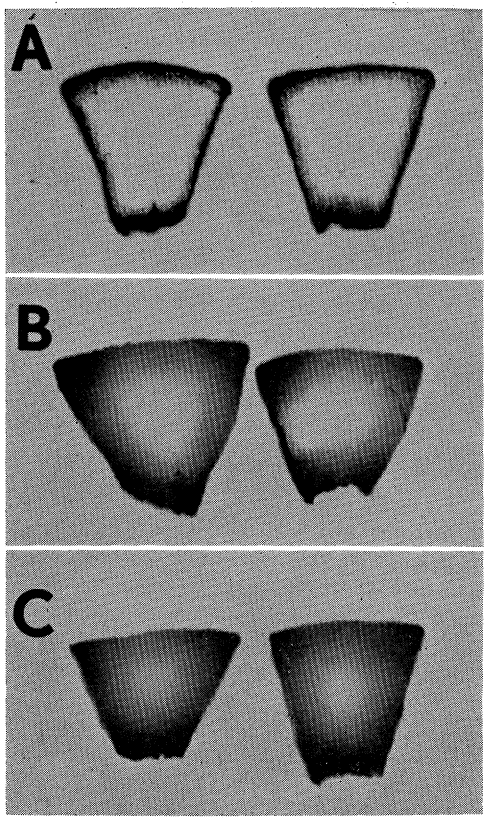

Fig. 1. Autoradiograms of transverse sections of mature Stayman apple slices submerged in a solution of $Ca^{45}Cl_2$ for 15 min at 68°F. A, submerged only; B, submerged and processed; C, submerged, subjected to vacuum and steam, and processed.

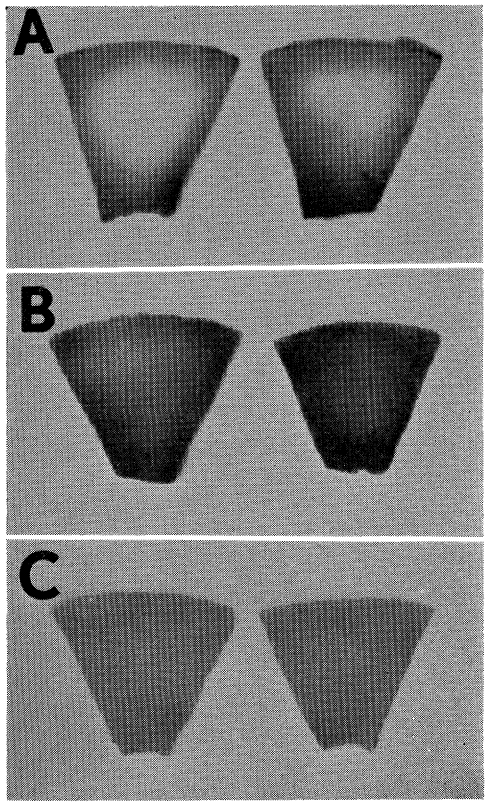

Fig. 2. Autoradiograms of transverse sections of immature Stayman apple slices submerged in a solution of $Ca^{45}Cl_2$. A, B, and C, respectively, depict 0, 5, and 15 min of vacuum. Following deaeration, slices were steamed and processed.

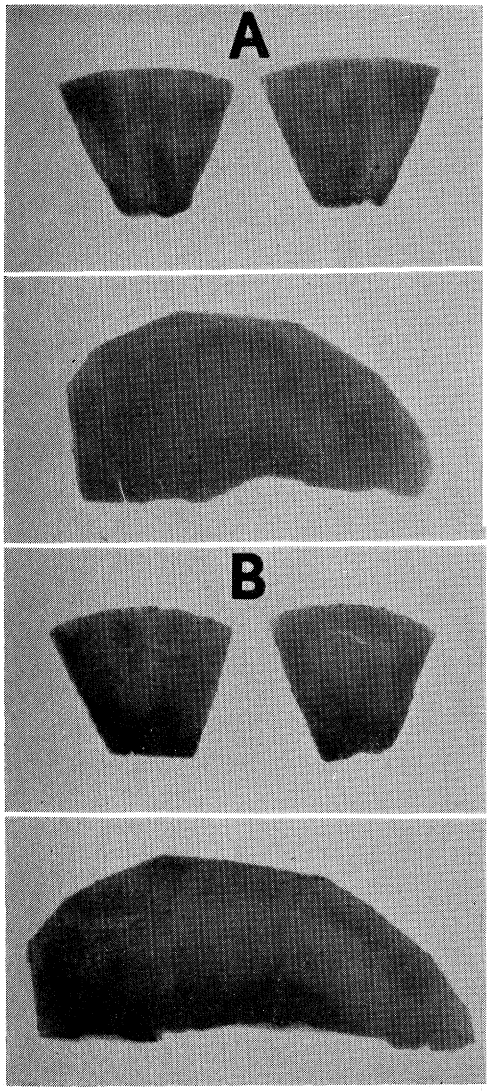

Fig. 3. Autoradiograms of transverse and longitudinal sections of overmature Stayman apple slices submerged in solution of $Ca^{45}Cl_2$ and deaerated. A, *deaerated 15 min;* B, *not deaerated. Slices were steamed and processed.*

"firmed," while the center would remain relatively soft. When slices were held in a Ca salt solution under a partial vacuum for 5 min, Ca penetrated deeper into the tissue, but light areas still appeared near the center area. A vacuum for 15 min allowed an apparent even distribution of Ca throughout the slices (*C*).

Treatments represented by prints in Fig. 3 compare the difference between deaerated and undeaerated slices from apples considered overmature. When the occluded gases were removed by vacuum, Ca was distributed evenly in the tissue (*A*). Failure to remove these gases resulted in their expansion to the point of rupturing the tissues (*B*). As a result of this rupture, the slices had a soft, mushy texture, although a considerable amount of Ca^{45} was present in the center of the slice. The Ca^{45} was carried to the innermost area through these breaks in the tissue. The light, wavy streaks, visible in the slices not deaerated, show the region where the rupture occurred.

Prints in Fig. 4 show that Ca salts are ineffective as a firming agent when applied as solution in the can in

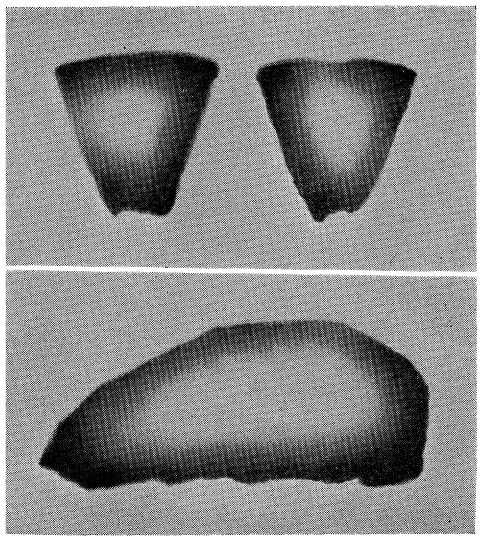

Fig. 4. Autoradiograms of longitudinal and transverse sections of mature Stayman apple slices subjected to vacuum and steam. Slices were processed in cans containing $Ca^{45}Cl_2$ solution.

which mature slices are processed. The results were similar to those obtained by the submersion procedures; only the outer tissues received the Ca ions.

Results of this work indicate that Ca salts cannot be applied effectively by methods of submerging or processing slices in Ca salt medium. The vacuum-pressure technique employed in this study proved most effective. A vacuum for 15 min was shown to be necessary to obtain an even distribution of Ca in the slices. Gases in the slice may prevent penetration of Ca to the center tissue or cause the tissue to rupture, resulting in a product with a mushy texture.

The procedure described may be employed to ascertain the efficiency of the canning process where plant tissues, fruits and vegetables, are treated with Ca salts.

Of the various techniques employed to determine penetration, autoradiography offers the advantage that one can visually ascertain the location of Ca ions within the tissue and get some idea of the relative concentrations. In relatively soft apple tissue the presence of Ca ions would indicate that the tissue was firmed. In slices used as controls (no $Ca^{45}Cl_2$ applied), the resultant autoradiograms showed no darkened areas.

The present investigation shows that the method employed to apply Ca salts to apple slices influences the depth of salt penetration and distribution in the tissue. Autoradiograms presented show how different treatment manipulations, such as submersion, deaeration, steam (pressure), and processing, influence the movement of Ca ions in apple slices.

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